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ZEOLITIC MATERIALS

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Zeolites can be defined as alumino-silicates with a three-dimensional framework structure with extra-framework cations and water molecules. They have since their discovery in the eighteenth century fascinated mineralogists. However, it was not until the advent of X-ray crystallography we got a full understanding of their properties and diversity. Crystallography still plays a crucial role in characterization of zeolites, and goes hand-in-hand with the development of new materials and new synthesis methods. The most important driving force in these developments is their use as catalysts. In particular, their use in petro-chemistry have made them one of the most important groups of chemicals today. Essentially all lighter fuels are produced using a zeolite cracking catalyst. These developments have led to a large diversity of compounds no longer restricted to alumino-silicate frameworks, but including a number of framework elements like for instance P and Ge, generally termed zeolitic materials.

As mentioned, X-ray crystallography has played and is still playing a crucial role in the characterization of zeolitic materials. Developments of methods to solve crystal structures from powder diffraction data has been very beneficiary to zeolitic materials as they are often difficult to grow as large enough single-crystals. The high angular resolution that can be achieved using synchrotron radiation has further augmented the use of powder diffraction for structure solution. The high intensity from synchrotron radiation has also enabled in situ studies of processes in zeolitic materials. New detectors allow time resolutions down to seconds and enable us to follow for instance synthesis, calcination, dehydration and deactivation in situ. With a time resolution of minutes, powder diffraction data suitable for Rietveld refinements can be obtained and structural developments can be followed in fine details.

The catalytic processes involving zeolites are heterogeneous and as such requires a large surface area. Although the internal channel surface area is very large, the outer surface area is still a limiting factor. To increase the outer surface area several methods are available, for instance producing nanosized crystals, post-synthetic treatments to introduce cracks and defects, and introducing mesopores during synthesis. For these nanostructured materials electron microscopy, SEM, TEM and electron diffraction, are necessary complementary tools for characterization.

The talk will give a short historical view of the importance of crystallography in the study of zeolitic materials and through a number of examples illustrate how the developments in methods and experimental facilities are giving opportunities for new types of crystallographic studies.